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United States Department of Agriculture,

BUREAU OF CHEMISTRY—Circular No. 26.

H. W. WILEY, Chief.

EXTRACTS FROM THE PROCEEDINGS OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS, 1905.

[An advance circular, giving the recommendations of referees as adopted, motions, and appointments affecting the work of 1906.]

REPORTS OF COMMITTEES ON RECOMMENDATIONS OF REFEREES.

COMMITTEE A.—J. P. STREET, CHAIRMAN.

(Phosphoric acid, potash, nitrogen, soils, ash, and insecticides.)

(1) Potash.

It is recommended—

1. That the study of the volumetric method be continued, with particular attention to its use in soil analysis.

Adopted.

2. That a method of incineration at low temperature and subsequent solution in dilute acid be examined and reported on at the next annual meeting.

Adopted.

3. (The association was asked to carefully consider the adoption as official of the following two methods coming over from the preceding year for final consideration, the referee for 1905 not giving these changes an unqualified indorsement):

(1) That the directions for making the solution in mixed fertilizers given under the determination of potash in Bulletin 46, page 21, be made to read as follows:

> Boil 10 grams of the sample with 300 cc of water plus 5 cc of hydrochloric acid for thirty minutes. Add a few drops of phenolphthalein and carefully neutralize with sodium hydrate free from potash, avoiding a large excess. Add sufficient powdered ammonium oxalate to precipitate all the lime present, cool, dilute to 500 cc, mix, and pass through a dry filter.

Motion to make this change official was lost.

(2) That the directions for the determination of moisture in potash salts, sodium nitrate, and sulphate of ammonia, given under the determination of moisture, Bulletin 46, page 11, be made to read as follows:

> In potash salts, sodium nitrate, and ammonium sulphate heat 2 grams at 130° in oil oven for 10 hours. The loss in weight is considered as moisture.

The motion to make this change official was lost.

(2) Phosphoric Acid.

It is recommended—

1. That the referee on phosphoric acid take up, for report at the next meeting of the association, methods applicable under American conditions to the official examination of basic slag phosphates. (Motion by Mr. Frear referred to committee.)

Adopted.

2. That the subject of an accurate determination of iron oxid and alumina in rock phosphates be examined by the referee on phosphoric acid and an official method be recommended to the association next year. (Motion by T. S. Gladding referred to the committee.)

Adopted.

3. That a number of chemists be requested to send to the referee on phosphoric acid samples of the citrate ammonia solution employed by them, and that the referee examine such samples as to neutrality and that such examination be reported to the chemists at the next annual meeting. (Motion by T. S. Gladding referred to the committee.)

Adopted.

(3) Nitrogen.

It is recommended—

1. That the study of availability by both the neutral and the alkaline permanganate methods be continued, particularly with the view to determining the amount of material to be used in the neutral method on commercial fertilizers. (Continued from 1905.)

Adopted.

2. That in the official Gunning method for the determination of nitrogen the addition of one-half to three-quarters of a gram of copper sulphate, after digesting with sulphuric acid and potassium sulphate for one-half hour, be studied by the referee. (Recommendation by F. D. Fuller referred, on motion, to the committee.)

Adopted.

3. At the close of the report of Committee A, Mr. Davidson made the following motion, which, as it involved a change in an official method, was referred to the referee on nitrogen for recommendation in 1906. The motion is as follows:

Recommendation for Standardizing the Hydrochloric Acid Solution.

It is recommended—

That the silver precipitation method (Bul. 46, Rev., p. 14), under "4. Determination of nitrogen," be changed to read as follows:

By means of a preliminary test with silver nitrate solution, to be measured from a burette, with excess of calcium carbonate to neutralize free acid and potassium chromate as indicator, determine exactly the amount of nitrate required to precipitate all the hydrochloric acid. To a measured and also weighed portion of the standard acid add from a burette one drop more of silver-nitrate solution than is required to precipitate the hydrochloric acid. Heat to boiling, cover from the light, and allow to stand until the precipitate is granular. Then wash with hot water through a Gooch crucible, testing the filtrate to prove excess of silver nitrate. Dry the silver chlorid at 140° to 150° C.

(4) Insecticides.

Three methods coming up for final action as to their adoption as official methods were first considered.

It is recommended—

1. That Methods I and IV of the four modifications of the Avery-Beans method of determining total arsenic in Paris green[a] be adopted as optional official methods.

Adopted as official.

2. * * * and that the thiosulphate method be adopted as an optional method using N/20 instead of N/10 thiosulphate solution.

Adopted as official.

3. That the Blank and Finkenbiener peroxid method be adopted as the official method of determining formaldehyde in strong solutions, and that the Romijn

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 90, pp. 96–97.

potassium cyanid method be adopted as an official method for analyzing dilute solutions.

Opposed by referee for 1905 and motion lost.

4. That the work on London purple be continued, special attention being given to the modifications proposed by Mr. Davidson for the removal of part of the color.

DAVIDSON'S MODIFICATION.

Total arsenious oxid.

Place 2 grams of London purple in a beaker and dissolve in about 80 cc of water and 20 cc of concentrated hydrochloric acid at a temperature of 80°. Cool and add sodium carbonate in slight excess, transfer to a 250 cc flask, and bring to the mark; shake and filter through a dry filter into a dry beaker; acidify 50 cc with hydrochloric acid and add sodium bicarbonate, titrating with iodin as usual.

Total arsenic oxid.

Acidify 50 cc of the solution, prepared as described under "Total arsenious oxid," with hydrochloric acid, add 25 cc of concentrated hydrochloric acid and 3 grams of potassium iodid. Then proceed as directed under this determination in Circular 10, Revised, Bureau of Chemistry, page 4.

Adopted.

5. That the hydrogen peroxid method for determining sulphur in sulphur dips and similar compounds be further tested, effort being made to have all analyses made simultaneously.

Adopted.

6. That the hydrogen peroxid method for determining formaldehyde be tested in its modified form.

Adopted.

7. That the methods of determining available chlorin in bleaching powder be further compared.

Adopted.

(5) SOILS.

It is recommended—

1. That the determination of water-soluble plant food contained in the soil be further investigated.

Adopted.

2. That further study be made of the use of fifth-normal nitric acid in the determination of available plant food.

Adopted.

3. That the use of ammonium sulphate for the removal of the last appreciable amounts of barium from the solution of total alkalies be thoroughly tested with a view to its incorporation in the official methods.

Adopted.

4. That the sodium peroxid method for ultimate analysis and the preliminary separation of phosphorus as iron phosphate in ammoniacal solution previous to its re-solution and precipitation as ammonium phospho-molybdate be further investigated.

Adopted.

(Recommendations 5 and 6 were made as the result of the following resolution offered by Mr. Davidson and referred to the committee:

Resolved, That Committee A on recommendations be requested to consider the question of size of mesh of sieve to be used in preparing samples of soil for analysis.)

5. That the third line, fourth paragraph, section 1, under "VI. Methods for the analysis of soils" (Bul. 46, p. 71), be changed to read "one millimeter," instead of

"one-half millimeter," and that the referee be requested to consider this matter in his work with the view to its adoption next year.

Referred to referee for 1906 for recommendation as to final action.

6. That on page 74, section *h*, first line (Bul. 46), the clause "passed through a sieve of one millimeter mesh," be omitted.

Referred to referee for 1906 for recommendation as to final action.

(6) ASH.

It is recommended—

1. That the provisional nitric-acid method for sulphur in plants be dropped.

Adopted.

2. That the peroxid method be adopted as a provisional method for sulphur in plants as follows:

Weigh out 15 grams of sodium peroxid (free from sulphates) and introduce about three-fourths of it at once into a 100 cc nickel crucible; add a little water and boil over a sulphur-free flame until excess of water is completely driven off. Allow to cool until pasty and stir 2 grams of material into it as quickly as possible; heat cautiously until danger of foaming has passed, adding the remainder of the peroxid in small portions from time to time to complete the oxidation. After fusion is complete, allow crucible to cool; dissolve contents in water, transfer to a beaker and add a slight excess of hydrochloric acid. Heat to boiling, filter if not perfectly clear; dilute to about 400 cc and add 10 cc of a 10 per cent solution of barium chlorid. Let stand over night, filter, and weigh the barium sulphate in the usual manner. Make blank test with the reagents.

Adopted.

3. That the referee for next year be requested to investigate the Barlow-Tollens method as a means of distinguishing between organic sulphur and sulphur of the ash.

Adopted.

(7) SEPARATION OF NITROGENOUS BODIES.

A. MILK AND CHEESE PROTEIDS.

It is recommended—

1. That this work be continued, and that special attention be given to the problem of making a complete extraction of water-soluble nitrogenous bodies and to the complete separation of these in filtering.

Adopted.

2. That the amount of water used in making the extract be increased from 500 cc to 1,000 cc.

Adopted.

3. That after the fat and insoluble nitrogenous bodies have been removed by the absorbent cotton, the filtrate be passed through asbestos.

Adopted.

B. SEPARATION OF VEGETABLE PROTEIDS.

It is recommended—

That the method of Horace T. Brown (Transactions of Guiness Research Laboratory, vol. 1, part 1, p. 61, 1903), on The Estimation in Barley and Malt of the Total Amount of Nitrogenous Substances Soluble in Water, be used for the separation of nitrogenous bodies in barley and malt.

Adopted as a provisional method.

C. SEPARATION OF MEAT PROTEIDS.

No recommendations reported to committee.

COMMITTEE B.—L. L. VAN SLYKE, CHAIRMAN.

(Dairy products, foods and feeding stuffs, sugar, tannin, and drugs.)

(1) SUGAR.

A. CHEMICAL METHODS OF SUGAR ANALYSIS.

It is recommended—

1. That the work as outlined in the report of the referee on sugars be continued until uniform methods are obtained for the various reducing sugars; that copper-sugar factors be determined for the various concentrations of the different reducing sugars and for various mixtures and proportions of sucrose and the reducing sugars.

Adopted.

2. That the methods selected by the international committee for unifying methods of sugar analysis, now provisional, be made official. (See Proceedings, 1902, pp. 58, 59, pars. 1 to 9, inclusive.)

Goes over for action in 1906.

3. That method (*a*) for the determination of copper in the cuprous oxid precipitate, requiring reduction in hydrogen (see Bul. 46, p. 37), be dropped as an official method of this association.

Goes over for action in 1906.

4. That methods (*c*) and (*d*) for the determination of copper in the cuprous oxid precipitate, requiring the electric deposition of the metal (see Bul. 46, p. 38), be dropped as official methods of this association.

Goes over for action in 1906.

5. That Low's thiosulphate method for determining the copper in the cuprous oxid precipitate be substituted for the volumetric permanganate method as a provisional method. (See Bul. 46, p. 38.)

Action deferred until 1906 by request of referee.

B. SPECIAL ANALYTICAL METHODS OF SUGAR ANALYSIS.

It is recommended—

1. That the work upon the separation of sugars next year be submitted to collaborative trial.

Adopted.

2. That the work upon the identification and determination of the various organic solids not sugar be continued.

Adopted.

C. MOLASSES ANALYSIS.

It is recommended—

1. That when the color of a molasses is so deep as to make difficult the decolorization of a "normal solution" to the extent necessary for accurate polarization, it should be made permissible—using proper precautions—to employ solutions of one-half or one-fifth the recognized normal concentration, and to obtain the actual polarization of the sample by multiplying the observed results by 2 or by 5.

2. That the present direction of the official method, "With molasses and massecuites add sufficient acetic acid to convert the (lead) subacetate into the neutral acetate," be so changed as to indicate that the addition of acid should be made to the *clarified filtrate intended for the direct polarization* and not to the basic acetate solution nor to the mixture of molasses and lead reagent previous to filtration.

3. That the use of boneblack, condemned by the international commission some years ago and now made unnecessary by the polarization in weaker solutions, be discarded.

4. That the association adopt, provisionally, the use of potassium oxalate as a

reagent for precipitating the excess of lead from clarified sugar solutions previous to subjecting them to the determination of reducing sugars.

5. That such a portion of the paragraph on p. 39, Bul. 46, revised edition of 1899, as relates to the preparation of molasses and massecuite solutions for polarization, be changed to read substantially as follows (following the words "precipitable matter" in line 9):

> With molasses and massecuites, the employment, for clarification, of excessive quantities of the lead reagent is to be avoided. In case that 5 cc of standard lead subacetate solution fails to clarify solutions of these materials, containing the normal weight in 100 cc, sufficiently for accurate polarization, it is advisable to make the solutions up to 200 cc or even to 500 cc, and to obtain the actual polarizations of the samples by multiplying the observed polarizations by 2 or by 5.[a]

The portion of the filtrate employed for the direct polarization should be made faintly but distinctly acid to litmus paper, shortly before polarization, by the addition of a minimum amount of glacial acetic acid.

Furthermore, it is recommended that the paragraph beginning on line 16, p. 39, Bul. 46, and relating to the temperature of polarization be extended substantially as follows:

> When solutions of half-normal or fifth-normal concentration are used for polarization, the deviation of the zero point of the saccharimeter should be established by at least five settings and readings—preferably with the polariscope tube in place, empty but fitted with its cover glasses[b]—and at least five settings and readings should be made for each polarization. In every case the mean observed polarization should be corrected, before multiplication, for the deviation of the zero point.

Recommendations 1 to 5 go over to 1906 for recommendations by referee and final action.

6. With regard to next year's work upon molasses analysis, it is recommended that further study be made of the choice of a constant or variable factor for employment in the Clerget equation, and that a table of logarithmic factors be prepared for use with the new modification of the Soxhlet solution, in computing the percentage of reducing sugars.

Adopted.

(2) FOODS AND FEEDING STUFFS.

The following recommendations were made by C. A. Browne, jr., associate referee on sugar, who says: "The extent to which this class of feeds is at present being adulterated renders an action of this character exceedingly desirable."

It is recommended—

1. That a committee be appointed by the association for the establishment of a set of standards for the composition of the various unmixed feeds—such as wheat bran, rice bran, cotton-seed meal, etc.—these standards to give the maximum or minimum percentages of protein, ash, fat, and crude fiber which these different unadulterated feeds should contain.

Referred to the food standards committee.

2. That the method adopted by the Fifth International Congress of Applied Chemistry[c] for the analysis of molasses feeds be adopted by the association. The method reads as follows:

> Twenty-five grams of the molasses feed are dried for three hours at 80° C.; after cooling the loss in moisture is determined and the residue thoroughly ground. Five

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[a] The darkest third molasses, when its normal weight is diluted to the volume of 500 cc, ought not require more than 12 to 13 cc of standard lead subacetate for satisfactory decolorization.

[b] Sawyer. The commercial analysis of cane molasses. J. Amer. Chem. Soc., 1905, *27:* 704.

[c] Bericht über die Arbeiten der Internationalen Kommission für die Analyse der Kunstdünger und Futtermittel, p. 27.

grams of the powdered feed are then freed from sugars by washing upon a filter paper or Gooch crucible with 100 cc of cold water, added drop by drop. The residue is then dried in a water oven and extracted with ether in the usual way.

Argument for recommendations.—The present official method for fat determination is not applicable to the molasses feeds, owing to the occlusion of fat by the dried residue of sugars. Our work at Audubon Park upon molasses feeds, which are very extensively manufactured and used in Louisiana, has demonstrated this fact conclusively. Although the constitution states (Article VII) that no changes shall be made in our official methods of analysis except by unanimous consent until the association has thoroughly tested such proposed change, I believe that the circumstances of the case, supported as they are by the authority of the international commission, should lead to the immediate adoption of this resolution.

Referred to referee for recommendation in 1906 as to final action.

(3) DAIRY PRODUCTS.

It is recommended that the referee for next year be requested—

1. To continue the study of the effect of preservatives, especially formaldehyde, mercuric chlorid, and carbon bisulphid, upon the determination of the proteids of milk.

Adopted.

2. To study methods for determining sugars in condensed milks and dried milks or milk powders.

Adopted.

3. To study methods of detecting the adulteration of butter with small quantities of foreign fats.

Adopted.

(4) TESTING OF CHEMICAL REAGENTS.[a]

The amount of information at present possessed by the committee is insufficient to warrant recommending any action relative to the adoption of standards for the chemicals so far investigated, but the committee does recommend that the work be continued and that the work of the succeeding committee be extended to include ten additional chemicals which in the opinion of the committee may be considered the most important.

Adopted.

(5) MEDICINAL PLANTS AND DRUGS (OPIUM ASSAYING).

It is recommended—

1. That this association adopt the method prescribed by the eighth revision of the United States Pharmacopœia, page 329, as a provisional method for gum opium, powdered opium, deodorized opium, and granulated opium.

2. That the work on the assay of opium be continued, utilizing such methods as appear to give satisfactory results, and that the work be extended to ipecac and golden seal.

Adopted.

3. That associate referees be appointed to take up different features of the work.

Adopted.

(6) TANNIN.

It is recommended that the following questions be made subjects of collaborative research during the ensuing year:

1. Soluble solids filtration, with especial reference to the time-contact method, so termed, in conjunction with the temperature problem.

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[a] Referred by error to this committee, together with recommendations on drugs, and acted on by the association at this time.

2. Analysis of liquors and the effect of the acidity of liquors upon the analysis.

3. Estimation of acid in tan liquors, with a view to improving the present method.

4. The influence of acidity and alkalinity upon the chroming of hide powder and upon the subsequent analysis. The elimination of the sulphate factor and the merits of a fully or partially chromed, prepared hide powder.

5. Extraction, for the purpose of confirming or rejecting the recommendations of the referee.

6. The Parker-Payne method of tannin analysis.

7. Estimation of nitrogen in leather and tan liquors.

Adopted.

It is further recommended that the following methods and processes be adopted provisionally:

8. The chroming of hide powder by the addition of the entire amount of chrome alum at one time.

9. Limiting the moisture content of wet chromed hide powder used for analysis between 70 and 75 per cent.

10. The analysis of liquors as recommended by the referee.

11. The form of apparatus known as the combined evaporator and dryer.

Adopted.

Finally, it is recommended that—

12. An official method be drawn up from the present method (Circular 10, revised, 1902), including the provisional methods recommended in this year's report. (Draft of such a method submitted with report.)

Action deferred to 1906.

At the close of the report of Committee B, the secretary presented a letter from a committee of the American Leather Chemists' Association, signed by W. H. Teas, H. C. Reed, and J. H. Yocum, making the following suggestions in regard to the relations existing between the two associations in regard to the tannin work. These suggestions were in brief as follows:

> 1. That the executive committee of the Association of Official Agricultural Chemists appoint a special committee on recommendations for the tannin section and that the association appoint the same referee and associate referee on tannin as are appointed by the American Leather Chemists' Association.
>
> 2. That the Association of Official Agricultural Chemists abandon the appointment of a referee and associate referee on tannin and consequently omit for the present any supervision of the tannin methods.

By motion the suggestions made in this letter were referred to the referee for 1906 for consideration.

COMMITTEE C.—A. L. WINTON, CHAIRMAN.

(Food adulteration.)

(1) Saccharine Products.

It is recommended—

1. That the methods of Circular 23, Bureau of Chemistry (Methods for the examination of maple products, Hortvet), be adopted as provisional.

Adopted.

(2) Vinegar.

It is recommended—

1. That the following words in Bulletin 65, page 68, be eliminated: "The presence of malic acid distinguishes cider vinegars, though the quantity is often small." * * * "If a precipitate be obtained, parallel tests with silver nitrate and barium chlorid to determine the absence of chlorids and sulphates should be made before the presence of malic acid be considered proved." (Referred to committee, on motion by Mr. Tolman.)

Adopted.

(3) Flavoring Extracts.

It is recommended—

1. That Leach's test for coumarin be adopted as a provisional method. (See Leach, Food Inspection and Analysis, p. 738.)

Adopted.

2. That the colorimetric ferrous sulphate method for determination of vanillin be adopted as a provisional method. (Ibid., p. 735.)

Adopted.

3. That Winton and Bailey's modification of the Hess and Prescott method for the determination of vanillin, coumarin, and acetamid be substituted for the present gravimetric methods. (J. Amer. Chem. Soc., 1899, *21:* 256; 1902, *24:* 1128; 1905, *27:* 719.)

Adopted.

4. That Leach and Lythgoe's refractometric method for the detection of methyl alcohol be adopted as a provisional method in the analysis of extracts. (J. Amer. Chem. Soc., 1905, *27:* 964.)

Adopted.

5. That the recommendations of the associate referee on flavoring extracts in regard to work for the following year be adopted:

(*a*) A thorough test of the present methods for the determination of citral in oil of lemon.

(*b*) Application to extracts of the methods for the detection of caramel as applied to whiskies.

(*c*) Application of a method for measuring the purity of extracts by means of the volume of their lead acetate precipitate.

(*d*) Effect of the presence of glycerin on the precipitation of the resins.

(*e*) Study of the crystalline forms of vanillin and coumarin.

Adopted.

(4) Dairy Products.

It is recommended—

1. That the following method for the determination of added water in milk be adopted as provisional:

DETECTION OF ADDED WATER (METHOD OF LEACH AND LYTHGOE).

To 100 cc of milk at a temperature of about 20° C. add 2 cc of 25 per cent acetic acid (sp. gr. 1.0350) in a beaker, and heat the beaker, covered with a watch glass, in a water bath for 20 minutes at a temperature of 70° C. Then place the beaker in ice water for 10 minutes and separate the curd from the serum by filtration through a 12.5 cm. plaited filter.

Transfer about 35 cc of the serum to one of the beakers that accompanies the control-temperature bath used in connection with the Zeiss immersion refractometer, the bath being of the type with openings in the top for 10 beakers. Place the beaker in one of the openings, use the ground-glass strip at the bottom of the bath, and by means of the regular refractometer heater or similar device maintain a constant temperature of exactly 20° C. in the water surrounding the beaker, using a delicate thermometer, reading to tenths of a degree. Immerse the end of the refractometer in the serum in the beaker, and when the temperature is exactly 20° C. take the reading on the scale.

If the temperature varies from 20° C., the reading may be calculated on that basis by means of a correction table. A reading below 39 indicates added water; between 39 and 40 the sample is suspicious.

Adopted.

(5) Spices.

It is recommended—

1. That the methods for the analysis of prepared mustard outlined by the associate referee for spices be adopted as provisional methods:

ANALYSIS OF PREPARED MUSTARD.

Sampling.—The solid portion of the material is commonly in a finely divided condition and does not require grinding; but as it tends to settle, leaving a more or less clear liquid on the surface, thorough mixing is absolutely essential. This may be accomplished by stirring with a spoon immediately before removing each portion for analysis.

Solids.—Dry 5 grams in a flat-bottomed platinum dish on a water bath until the mixture appears dry and finally dry to constant weight at 100° C. in a water oven.

Ash.—Burn the dry residue obtained in the determination of water to a white ash in a muffle furnace at a heat below redness.

Salt.—Determine chlorin in the ash with silver nitrate either gravimetrically or volumetrically, using potassium chromate as an indicator.

Ether extract.—In a capsule place 10 grams of the material, about 30 grams of sand, and a short stirring rod. Heat on a water bath with constant stirring to apparent dryness and finish the drying in a water oven. Grind until all the lumps are broken up and extract for 16 hours in any suitable apparatus with absolute ether. Dry the extract at 100° C. and weigh.

Protein.—Determine nitrogen by the Kjeldahl method and multiply the results by 6.25.

Crude fiber.—Weigh 8 grams of the material (equivalent to about 2 grams of dry matter) directly into an Erlenmeyer flask and proceed as with spices,[a] except that care is taken to add at first only a small amount of 1.25 per cent acid or alkali and shake thoroughly until all lumps are broken up. If this precaution is not taken, the lumps will resist the action of the acid or alkali and the results will be high. Extraction of the fat previous to the treatment is impracticable, as this necessitates preliminary drying, after which the material forms a horny mass.

This method is valuable as furnishing indications as to whether the product is made from ground mustard seed, mustard flour (decorticated ground mustard seed), or mustard hulls, comparisons being made of the results calculated to a water-fat and salt-free basis.

Crude starch.—(Reducing matters by direct inversion calculated as starch.) Proceed as with spices,[b] except that 10 grams of the material are treated directly with 200 cc of water and 20 cc of 25 per cent hydrochloric acid without previous washing or extraction, and the solution is made up to 250 cc before filtering and drawing off the aliquot.

This method is of value in detecting starchy adulterants, although the results obtained are higher than the true percentage of starch, owing to the conversion of constituents of the mustard seed into copper-reducing substances and the frequent presence of sugar. As the starchy matter used is often in the form of paste, preliminary washing with water is impracticable.

Acidity.—Weigh out 10 grams into a 200 cc graduated flask, make up to the mark with water, shake, filter through a dry paper, and determine the acidity in 100 cc by titration with N/10 potassium hydrate solution, using phenolphthalein as an indicator.

Tests for dyes.—Test for turmeric by the boric-acid method or by microscopic examination.[c]

Examine for coal-tar colors by Arata's or Sostegni and Carpentieri's method,[d] with subsequent examination of the dyed wool. Turmeric also imparts a dull yellow color to the wool, but this color is changed to brown-red with alkali and does not give marked color reactions with concentrated hydrochloric or sulphuric acids. Nitro-colors dye the wool brilliant shades of yellow, which on treatment with hydrochloric acid are partially bleached (salts of dinitrocresol), or remain unchanged (tartrazin, etc.). The azo colors commonly used are changed to crimson or violet on addition of strong acid to the dyed fabric or the filtered aqueous solution of the original material.

Tests for preservatives.—Test for boric, salicylic, and benzoic acids by the usual methods.[e] As shaking with ether dissolves a large amount of fat, salicylic or benzoic acid should be separated from this fat by sublimation, solution in dilute alcohol, or conversion into the ammonium salt and solution in water. These acids may also be obtained from the original material by distillation.

Adopted.

(6) Fats and Oils.

It is recommended—

1. That the Hanus method, as given in the Proceedings of 1903,[f] be adopted as official for the determination of the iodin absorption of oils and fats.

Adopted.

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[a] U. S. Dept. Agr., Bureau of Chemistry, Bul. 65, p. 58.

[b] Ibid., p. 57.

[c] Ibid., p. 120.

[d] Ibid., p. 112.

[e] Ibid., pp. 108–110.

[f] Ibid., Bul. 81, pp. 63–64.

2. That the method for the titer test be adopted as provisional. (See Circular 27, Bureau of Chemistry, U. S. Dept. of Agr.)

Adopted.

3. That the following recommendations for work for next year be adopted:

(*a*) Study of methods for determination of moisture and impurities in fats.

(*b*) Study of method for making total fatty acid determination in soap stock.

(*c*) Study of method for cold test.

(*d*) Further study of drying of fatty acids in the titer test.

(*e*) Further study of the Belfield test.

Adopted.

4. That the recommendation made at the association meeting in 1903 (Bul. 81, p. 64), for changing the correction for temperature for index of refraction from 0.000176 to 0.000365 now be made official. (Bul. 46, revised, p. 31.)

Adopted.

(7) Preservatives.

It is recommended—

1. That Arnold and Mentzel's method for the detection of formaldehyde (using phenyl-hydrazin hydrochlorid and ferric chlorid) be adopted provisionally. (Bul. 90, p. 47, Method 5; Zts. Nahr. Genussm., 1902, *5:* 353.)

2. That Arnold and Mentzel's method for the detection of formaldehyde (using phenyl-hydrazin hydrochlorid and potassium ferricyanid) be adopted provisionally. (Bul. 90, p. 48, Method 6; Chem. Ztg., 1902, *26:* 246; abs. J. Chem. Soc. (Lond.), 1902, (2), *82:* 367; abs. Chem. Centrbl., 1902, pt. 1, p. 1077.)

3. That the Rimini test for the detection of formaldehyde be adopted provisionally. (Bul. 90, p. 48; Method 7; Ann. di Farmacal., 1898, p. 97; abs. Chem. Centrbl., 1898, pt. 1, p. 1152; abs. J. Soc. Chem. Ind., 1898, *17:* 697.)

4. That the colorimetric method for the determination of salicylic acid be adopted as a provisional method. (Compt. rend., 1881, p. 278.)

5. That the methods suggested by the referee for the quantitative determination of salicylic acid, benzoic acid, and saccharin by weighing and titrating the residue from extraction and sublimation (after previous separation by distillation when necessary) be adopted provisionally. (Bul. 90, p. 59, Methods 2, 3, and 4.)

6. That the Blarez method for the detection of fluorids, as modified by Leach, be adopted as a provisional method. (Chem. News, 1905, *91:* 39; Ann. Rept. Mass. State Bd. of Health, 1905.)

7. That the method for the detection of fluorids, borfluorids, and silicofluorids directly from the ash be provisionally adopted. (Bul. 59, p. 63.)

8. That the rearrangement of the provisional methods for the detection of preservatives suggested by the referee be adopted.

Recommendations 1–8 were adopted.

(8) Distilled Liquors.

It is recommended—

1. That the Trillat test for the detection of methyl alcohol in distilled liquors be added to the provisional methods. (Analyst, 1899, *24:* 13, 211–212.)

2. That the Riche and Bardy test for the detection of methyl alcohol in distilled liquors be added to the provisional methods. (Allen's Commercial Organic Analysis, 3d ed., *1:* 80.)

3. That the method of Leach and Lythgoe for the estimation of ethyl and methyl alcohol in mixtures of the same be adopted as a provisional method. (This mixture is modified for different liquors, and the following is more specifically for whiskies):

Fifty cc of the sample are weighed and mixed with 75 cc of water and 100 cc distilled off. The distillate is weighed, specific gravity taken, and calculation of total alcohols made for weight of sample, using ethyl alcohol tables. The distillate is then examined by the immersion refractometer and the reading corrected to 20° C.. using per cent alcohol, by weight (as total alcohol). Find from Table II[a] the scale reading for ethyl and methyl alcohol corresponding to the per cent of alcohol, by weight, determined by specific gravity, and calculate as follows:

(Reading for ethyl)—(Reading of distillate) $= X$.

$X \div$ (difference between ethyl and methyl readings) $100 =$ per cent of the alcohol present which is methyl.

Then calculate to weight or volume in original sample.

4. That the following method for the determination of ethereal salts be substituted for the method given in Bulletin 65, page 98:

In 50 cc of the distillate neutralize the free acid with N/10 alkali, using phenolphthalein as an indicator, then add 25 cc N/10 alkali and either heat for one hour with a reflux condenser, cool and titrate with N/10 acid, or allow the solution to stand over night in a stoppered flask with the excess of alkali, then heat with a reflux condenser for one-half hour at a low temperature, cool and titrate. The number of cubic centimeters of N/10 alkali used in the saponification of the esters is calculated as ethyl acetate.

The distillate for esters, aldehydes, and furfurol is prepared in the following manner: Add 25 cc of water to 200 cc of the whisky and distill until 200 cc are distilled off. It is advisable to use a mercury valve in order to prevent loss of alcohol, and the distillation should be made slowly.

5. That the following method for the determination of aldehydes be substituted for the method given in Bulletin 65, page 97:

Determine the aldehydes in the distillate prepared for the esters. Five to ten cc of the distillate are diluted to 50 cc with aldehyde-free alcohol (50 per cent by volume), 25 cc of the reagent added, and allowed to stand for 20 minutes at 15° C. The solutions and the reagents should be at 15° C. before they are mixed.

Reagent.—Dissolve 0.5 gram of pure fuchsin in 500 cc of water, then add a water solution of sulphur dioxid, equivalent to 5 grams of sulphur dioxid, and make the whole up to 1 liter. This solution can be used as soon as it becomes colorless and will keep for several days.

Aldehyde-free alcohol is prepared by treating ordinary 95 per cent alcohol with potassium hydroxid and distilling. Then this distillate is treated with about 3 grams to the liter of metaphenelenediamin hydrochlorate, and heated with a reflux condenser for several hours, then distilled slowly, not keeping the first 100 cc nor the last 100 cc. This will give an alcohol which will give only the slightest tinge of color when treated with the reagent.

The standard aldehyde solution is treated in the same way as the distillate, and the color of the standard must be very close to the color of the sample.

6. That the following method for the determination of furfurol be substituted for the method given in Bul. 65, page 98:

From 10 to 20 cc of the distillate are diluted to 50 cc with furfurol-free alcohol (50 per cent by volume). To this add 2 cc of colorless anilin and one-half cubic centimeter of hydrochloric acid (sp. gr. 1.125) and keep for 15 minutes in a water bath at about 15° C.

Prepare standards of known strength in the same way:

7. That the following method for fusel oil be given a trial by the association:

To 100 cc of whisky add 20 cc of N/2 sodium hydroxid, and then saponify the mixture by boiling for 1 hour under a reflux condenser.

The flasks are connected with a distilling apparatus and 90 cc distilled over, 25 cc of water added, and the distillation continued until 25 cc more come over.

To this distillate is added as much finely ground salt as it will readily dissolve and then saturated salt solution until the specific gravity reaches 1.10. The volume at this point is not much in excess of 200 cc.

The brine solution is extracted four times with carbon tetrachlorid, using 40, 30, 20, and 10 cc, and the carbon tetrachlorid washed three times with 50 cc of saturated salt solution. It is then run into a flask containing 1 cc of sulphuric acid, 10 cc of water, and 5 grams of potassium dichromate, and boiled for 8 hours under a reflux condenser.

When the oxidation is complete the flask is attached to the distilling apparatus after the addition of 30 cc of water and the contents distilled until but 20 cc remain.

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[a] J. Amer. Chem. Soc., 1905, *27*: 969.

Eighty cc more of water is then added and the distillation continued until but 5 cc are left. The distillate is neutralized to methyl orange and titrated with phenolphthalein, each cubic centimeter of N/10 sodium hydroxid equaling 0.0088 gram of amyl alcohol.

Notes.—Rubber stoppers can be used in the saponification and first distillation, but corks covered with tinfoil must be used in the oxidation and second distillation. Corks and tinfoil must be renewed frequently.

Instead of the saponification as given, the 100 cc of liquor may be allowed to stand over night on 20 cc of N/2 sodium hydroxid, and then distilled directly.

It is important to have the specific gravity of the mixture of brine and alcoholic distillate up to 1.10.

It is very difficult to make good joints where corks are covered with tinfoil, and it is a good plan to mark the height of the liquor on the flask in order to note any loss.

Carbon tetrachlorid must be purified by oxidation over acid bichromate for several hours with distillation and redistillation over barium carbonate.

8. That the following test be tried for artificial coloring matter in whisky:

Color insoluble in water.—Evaporate 50 cc of whisky to dryness on steam bath. Add a small amount of hot water to residue, filter through a small paper into a 50 cc flask, wash out all soluble color with water, using less than 25 cc, add 25 cc 95 per cent alcohol, cool, fill to mark, and mix. Compare the color with that of original whisky and record results in per cent color lost. The character of the residue left on evaporation both before and after adding water should be carefully noted.

9. That for the next year's work the referee make a further study of tests for artificial coloring matter and of the determination of fusel oil.

Recommendations 1–9 adopted.

(9) General.

On motion by Mr. Frear, as amended by Mr. Wiley, the following resolution was passed:

Resolved, That the referee on food adulteration be requested to provide next year for a systematic test of the method of Benedict and Manning[a] and of Maquenne's[b] method for determining hygroscopic moisture.

To provide for this work Committee C recommended that an associate referee for the determination of water in foods be appointed, and the recommendation was adopted.

GENERAL MOTIONS.

B. B. Ross. *Resolved*, That the fertilizer control officials be urged to require inspectors to use in sampling the method recommended in the report of the uniform legislation committee published in the Proceedings of the fifteenth meeting of the association.[c] The method is the same as that adopted by the Hot Springs meeting of commissioners of agriculture, and is now in use in many States.

Referred to committee on fertilizer legislation.

R. J. Davidson. *Resolved*, That the question of presentation of all papers, either by referees or others, be referred to the executive committee for arrangement as to the manner and time of presentation and the time for discussion of the same.

M. A. Scovell. *Resolved*, That the place of meeting for the next convention of the association be made discretionary with the executive committee.

A brief paper by Mr. R. E. Rose, State chemist of Florida, was referred to the committee on fertilizer legislation for the purpose of obtaining an expression of opinion on the following points:

(1) The limit of variation allowable in fertilizers; (2) to what extent the excess of one ingredient should be allowed to offset a deficiency in another, if at all; (3) what uniform allowance shall be made for differences between analysts working on the same sample—is not the present allowance of 0.20 rather broad for small percentages?

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[a] Amer. J. Phys., 1905, *13:* 309.

[b] Compt. rend., 1902, *135:* 208; 1905, *141:* 80.

[c] U. S. Dept. Agr., Bureau of Chemistry, 1899, Bul. 56, p. 131.

OFFICERS, REFEREES, AND COMMITTEES OF THE ASSOCIATION OF OFFICIAL AGRICULTURAL CHEMISTS FOR THE YEAR 1906.

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Mr. Harry Snyder, St. Anthony's Park, Minn.
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Nitrogen:
  Determination of nitrogen: J. H. Gibboney, Blacksburg, Va.
  Separation of nitrogenous bodies: R. Harcourt, Guelph, Canada (milk and cheese proteids).
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  Determination of nitrogen: C. L. Penny, Newark, Del
  Separation of nitrogenous bodies—
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Potash: B. B. Ross, Auburn, Ala.
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  (3) Fruit products: H. C. Lythgoe, Boston, Mass.
  (4) Wine: Julius Hortvet, St. Paul, Minn.
  (5) Beer: H. E. Barnard, Indianapolis, Ind. (General associate on food adulteration.)
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  (12) Fats and oils: L. M. Tolman, Washington, D. C.
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  (20) Preservatives: W. L. Dubois, Washington, D. C.
  (21) Determination of water in foods: F. C. Weber, Washington, D. C.
Sugar:
  Molasses methods: J. E. Halligan, Baton Rouge, La.
  Chemical methods: L. S. Munson, Washington, D. C.
Tannin: F. P. Veitch, Washington, D. C.
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Definition of "Plant Food."

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